

A composition for covering or filling cavities and sealing joints

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Abstract of GB975587

A self-hardening agent for filling holes, cracks and joints in concrete, stone or mortar comprises a mixture of monomeric and polymeric methyl methacrylate, a catalyst for the polymerization of methyl methacrylate and an inorganic filler, the surface area of the filler being from 30 to 100 sq. m. per kg. of polymerizable mixture. Fillers specified are quartz sand, felspar sand, glass fibres, asbestos talc, black iron oxide, titanium dioxide and heavy spar. As a catalyst there is specified a mixture of benzoyl peroxide and dimethyl-p-toluidine. Other specified ingredients of the polymerizable compositions are paraffin wax, wood turpentine oil and dibutyl phthalate.

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PATENT SPECIFICATION

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COMPLETE SPECIFICATION

A Composition for Covering or Filling Cavities and Sealing Joints

We, CHEMISCHE FABRIK GRUNAU G.m.b.H. of 9, Weissfrauenstrasse, Frankfurt, Main, Germany, a body corporate organised under the laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a self-hardening agent which is intended for immediate use and which is suitable for covering and filling cavities, holes and cracks in a substratum. Examples of the substratum to which the composition may be applied are mixtures of sand, shingle or gravel with hydraulically-set structural materials, for example mortar and concrete, such as are used for making roads, runways or factory floors. The substratum can also be natural stone.

Such mixtures can also be used to seal or fill joints of all types, such as movement and expansion joints of roadways and runways. Such sealing compositions have the advantage that they can satisfactorily be shaped, and are resistant to ageing and to fluctuations in temperature so that they do not breakup with changes in weather and have a very good adhesion after hardening. The agent consists of a mixture of monomeric and polymeric methyl methacrylate, an inorganic filler, such as sand, shingle or gravel and a catalyst for the polymerisation of methyl methacrylate. The surface area of the filler is from 30 to 100 m² per kilogram of polymerisable mixture.

The mixture of monomeric and polymeric methyl methacrylate can be obtained by partially polymerising monomeric methyl methacrylate, stopping the polymerisation when enough polymer has formed. The degree of polymerisation can easily be controlled by quenching polymerisation when a suitable viscosity is reached. The mixture can also be

made by dissolving polymeric methyl methacrylate in the monomer. The mixture should preferably contain 25 to 65 parts by weight of polymeric methyl methacrylate to 100 parts by weight of monomer. Such mixtures have usually the most suitable viscosity.

It is advisable to use quartz sand as the inorganic filler, containing as little moisture as possible. For special purposes, the fillers can also contain or consist of glass fibres, can also contain or consist of glass fibres, Coloured fillers, such as black iron oxide, titanium dioxide and heavy spar can also be employed, alone or mixed with sand. The quantity of filler can be varied within comparatively wide limits. Usually, 100 to 400 parts by weight of the inorganic filler are added to 100 parts by weight of polymerisable mixture.

100 to 300 parts by weight of filler are preferably used to 100 parts by weight of polymerisable mixture for joint-filling compositions, but this quantity depends upon the joint and the method of working. With very narrow joints, a comparatively-thin sealing composition must be used, but with wider joints, the composition can be more viscous, i.e. contain more fillers.

Not only the quantity but also the surface area of the filler is important. If a filler with too large a total surface area is used, the mixture of polymeric and monomeric methyl methacrylate will, sometimes, not be completely cured, and, in certain circumstances, may remain completely soft. If the filler has too small a surface, very high reaction temperatures may be reached: the resin may shrink and bubbles or cracks can be formed on hardening. The total surface of the fillers should be not more than 100 m² and not less than 30 m² for each kilogram of polymerisable mixture.

For certain purposes, especially if the mixture is to be applied as a thin layer with

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a large surface, it is advantageous to add a substance which lowers the viscosity of the mixture, which prevents the formation of a surface skin on hardening and makes more easy mixing, application and smoothing of the composition. Such substances are compounds of low viscosity which lengthen the processing times and reduce the resistance to deformation of the unpolymerized mixture. It is generally advisable to use 10 to 80, preferably 10 to 30, parts by weight of the substance which lowers the viscosity to 100 parts by weight of polymerisable mixture. The wood turpentine oil, obtainable under the name "Pineoil" is suitable for this purpose. The composition does not stick to the spreading apparatus and can easily be distributed by such conventional methods as the use of a scraper, a board or a trowel, so as to produce a closed surface which will not crack during the operation.

Mixtures which have proved most suitable for sealing joints are those which contain 20 to 80 parts by weight of the plasticiser to 100 parts by weight of the polymerisable mixture. The greater the content of plasticiser, the more elastic are the compositions when cured.

To prevent skin-formation and to improve the strength of the bond with the substratum, it is advisable to add, to the mixture, small quantities of wax; a hard paraffin wax with a high melting point is suitable for this.

The best catalysts are organic diperoxides or hydroperoxides, advantageously mixed with a plasticiser or solvent of high boiling point so as to make incorporation of the catalysts into the mixture more easy.

The amount of catalyst used depends upon its nature and upon the composition of the mixture. Generally speaking, less catalyst is required at higher external temperatures than at such lower temperatures as 0 to 5° C.

It is advantageous to add a polymerization accelerator to the mixture so as to accelerate the final hardening or curing. Preferable polymerisation accelerators are tertiary amines, advantageously with an aromatic radical directly linked to the nitrogen. To obtain short curing times, it is desirable to use catalyst and accelerator in substantially equivalent quantities.

The invention concerns also a method of covering and filling cavities, holes and cracks in a substratum. The procedure in this case is to mix the monomeric and polymeric methacrylates, which can already contain the accelerator and the substance lowering the vis-

cosity, with a catalyst which causes polymerisation of the monomeric methyl methacrylate, to add a filler to this mixture and, immediately it is produced, to cover the substratum with it or to introduce it into the cavities, holes or cracks which are to be filled. The mixture then hardens in a short time without being heated, even when the temperature is below 15° C.

It is also possible however, first to incorporate the filler into the methyl methacrylate and to add the catalyst last of all.

In order that the covering composition may be given a rough surface, it is possible while it is still liquid or soft or scatter sand or gravel on it. The material thus scattered unites firmly with the synthetic plastic composition during hardening, without being rolled in or without the surface being consolidated.

It is often advisable, especially when working with compositions with a high plasticiser content, to clean well the surfaces of the joints and to provide them with an preliminary coating before introducing the sealing composition. The previously described mixture of monomeric and polymeric methyl methacrylate, without plasticiser and without filler, can be used for such preliminary coatings.

Filling and coating compositions, prepared by the process of the invention, are easily worked, harden rapidly, shrink only slightly and show excellent bonding strength. The fillings themselves are highly resistant to mechanical and chemical stresses, due for example to de-icing salts, fuels and oils, and to weather influences. Consequently, they are particularly suitable for roadways, runways and factory or storehouse floors.

EXAMPLE 1

The following procedure was used to repair a concrete road.

The surface was cleaned with a hard brush. Cracks, holes and cavities were then filled with a mixture prepared by mixing 30.0 parts by weight of a mixture of monomeric methyl methacrylate containing 30% by weight of polymeric methyl methacrylate, 0.15 part by weight of hard paraffin wax, 0.8 part by weight of a 10% by weight solution of dimethyl-*p*-toluidine in methyl methacrylate and finally, adding a solution of 0.4 part by weight of dibenzoyl peroxide in 0.4 part by weight of dibutylphthalate.

To this mixture were added:

115	4 parts by weight of quartz sand	with a grain size from 0 to 0.2 mm.
16	" " " " " "	" " " " " " 0.06 to 0.4 mm.
22	" " " " " "	" " " " " " 0.4 to 0.6 mm.
24	" " " " " "	" " " " " " 0.6 to 0.8 mm.
and		
120	39 " " " " " "	" " " " " " 1.0 to 2.0 mm.

- The thickly liquid mixture could easily be poured into the holes, cavities and cracks and cured in 30 minutes to 1½ hours. After curing, a coating-composition was made by mixing
- 5 30.0 parts by weight of a mixture of monomeric methyl methacrylate with 30% by weight of polymeric methacrylate, 0.15 part
- 15 16.0 parts by weight of quartz sand with a grain size of 0 to 0.2 mm.
- 57.0 " " " " " " " " " " " " 0.06 to 0.4 mm. and
- 32.0 " " " " " " " " " " " " 0.004 to 0.6 mm.

were incorporated. The pasty mixture was poured on to the surface of the concrete slabs and distributed. Before it was cured some coarse sand was scattered on it to give a rough surface.

- 20 The coating was cured after 1 to 1½ hours, and traffic could travel over the surface immediately.
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EXAMPLE 2

- A joint-sealing composition was made by mixing 100 parts by weight of a syrup containing 70% by weight of monomeric methyl methacrylate and 30% by weight of polymeric methyl methacrylate with 0.5 part by weight of hard paraffin wax, 2.5 parts by weight of a 10% by weight solution of dimethyl-*p*-toluidine in monomeric methyl methacrylate, 43 parts by weight of industrial Pineoil and a paste made from 1.5 parts by weight of dibenzoyl peroxide and 1.5 parts by weight of dibutyl phthalate, 155 parts by weight of talc having a surface area of 80 m² per kilogram of polymerisable mixture were incorporated into this mixture.
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- The composition was cast to form prisms in accordance with DIN 1164 and left to harden for 5 days at 20°. On being compressed, a working load strength of 10 to 30 kg/cm² was found. The test elements with a thickness of 40 mm. could be compressed to a thickness of 28 mm. without any cracks forming. On removing the pressure, the test elements assumed their original shape within 12 hours. In the bending tensile strength test, the test elements were bent without breaking. After being subjected to the action of the bending load, the elements had a bending angle of about 60°, which was reduced to 10° about 12 hours after the load was removed.
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EXAMPLE 3

- 60 If the procedure according to Example 2 is used, but with 60 parts by weight of industrial Pineoil to 100 parts by weight of the methyl methacrylate mixture, much softer and more plastic elements are obtained. It was no longer possible to test the bending tensile strength by the method of DIN 1164.
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EXAMPLE 4

- 70 100 parts by weight of a mixture of monomeric methyl methacrylate with 30% by

weight of hard paraffin wax, 6.0 parts by weight of wood turpentine oil and 0.8 part by weight of a 10% by weight solution of dimethyl-*p*-toluidine in methyl methacrylate. Finally a solution of 0.4 part by weight of dibenzoyl peroxide in 0.4 part by weight of dibutylphthalate was added. Then

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weight of polymeric methyl methacrylate were mixed with 0.5 part by weight of hard paraffin wax and 0.25 part by weight of a 10% by weight solution of dimethyl-*p*-toluidine in methyl methacrylate. To this was added a paste of

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1.5 parts by weight of dibenzoylperoxide in

1.5 parts by weight of dibutylphthalate, and

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300 parts by weight of quartz sand with a grain size of 0.1 to 0.3 mm. were incorporated into the mixture.

The mixture was placed in moulds. After 5 days, the bending tensile strength and the compressive strength were determined. The bending tensile strength was 324 kg./cm.² and the compressive strength was 1080 kg./cm.².

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EXAMPLE 5

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When the procedure of Example 4 was followed, replacing the quartz sand by 700 parts by weight of feldspar sand with a surface area of 50 m² per kilogram of polymerisable mixture, elements which have a bending tensile strength of 226 kg./cm.² and a compressive strength of 923 kg./cm.² were obtained.

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WHAT WE CLAIM IS:—

1. A self-hardening agent intended for immediate use for sealing or filling joints for covering or filling cavities, holes and cracks in a substratum of hydraulically-set structural materials mixed with sand, shingle, gravel or natural stone, which comprises a mixture of monomeric and polymeric methyl methacrylate, a catalyst for the polymerisation of methyl methacrylate and an inorganic filler, the surface area of the filler being from 30 to 100 m.² per kilogram of polymerisable mixture.

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2. An agent as claimed in claim 1 which contains 25 to 65 parts by weight of polymeric methyl methacrylate to 100 parts by weight of monomeric methyl methacrylate.

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3. An agent as claimed in claim 1 and 2 in which the catalyst, preferably an organic diperoxide or hydroperoxide, is mixed with a plasticiser or solvent of high boiling point.

4. An agent as claimed in claims 1 to 3 which contains also a substance for lowering the viscosity of the mixture.

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5. An agent as claimed in claim 1 to 4

which contains 10 to 80 parts by weight of the substance for lowering the viscosity of the mixture to 100 parts by weight of polymerisable mixture.

- 5 6. An agent for filling joints as claimed in claims 1 to 5 which contains 20 to 80 parts by weight of the substance for lowering the viscosity of the mixture to 100 parts by weight of polymerisable mixture.

- 10 7. An agent for covering surfaces as claimed in claims 1 to 5 which contains 10 to 30 parts by weight of the substance for lowering the viscosity of the mixture to 100 parts by weight of polymerisable mixture.

- 15 8. An agent as claimed in claims 1 to 7 in which the substance for lowering the viscosity of the mixture is wood turpentine oil.

- 20 9. An agent as claimed in claims 1 to 8 which contains small quantities of wax or paraffin wax.

- 25 10. An agent as claimed in claims 1 to 9 which contains a polymerisation accelerator, preferably a tertiary amine, advantageously one which contains an aryl group linked directly to the nitrogen.

- 30 11. An agent as claimed in claims 1 to 10 which contains finely granulated inorganic fillers.

12. An agent as claimed in claims 1 to 11 which contains 100 to 400 parts by weight of fillers to 100 parts by weight of polymerisable mixture.

- 35 13. An agent as claimed in claims 1 to 12 in which the filler is quartz sand or feldspar.

14. An agent as claimed in claim 1 substantially as described with reference to any of the Examples.

- 40 15. A method of sealing joints in which an agent as claimed in claims 1 to 6 and

8 to 15 is applied immediately it is produced and is cured without supply of heat.

16. A method as claimed in claim 15 in which a catalyst is added to a mixture of monomeric and polymeric methyl methacrylate, a substance for decreasing the viscosity of the mixture and a polymerisation accelerator, then fillers are incorporated and the mixture is immediately applied.

17. A method as claimed in claim 15 and 16 in which the surfaces of the joint are given a preliminary coating before the agent is introduced.

18. A method as claimed in claims 15 to 17 in which a mixture comprising monomeric and polymeric methyl methacrylate and a catalyst is used as the preliminary coating.

19. A method of covering and filling cavities, holes and cracks in a substratum in which an agent as claimed in claims 1 to 5 and 7 to 14 is applied immediately it is produced and is cured without heating.

20. A method as claimed in claim 19 in which a catalyst is added to a mixture of monomeric and polymeric methyl methacrylate, possibly containing a substance for lowering the viscosity of the mixture and/or a polymerisation accelerator, then fillers are incorporated and the mixture is immediately applied.

21. A method as claimed in claims 19 and 20 in which sand or gravel is scattered on the surface of the agent immediately it is applied.

22. A process as claimed in claims 19 to 21 substantially as described with reference to Example 1.

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